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Technical Report

Metallurgical and mechanical characterization of stir cast $AA6061-T6-AlN_p$ composite

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ARTICLE INFO

Article history: Received 12 January 2012 Accepted 20 March 2012 Available online 30 March 2012

ABSTRACT

An attempt has been made to manufacture aluminium (6061-T6) matrix composites reinforced with aluminium nitride particles (AlN_p) of size 3–4 μ using indigenously developed modified stir casting method with bottom pouring arrangement in controlled argon environment. To improve the wettability of AlN_p with molten Al matrix, 2% Mg has been added. AA6061–AlN_p composites have been successfully produced at different weight percentages (viz 0, 5, 10, 15 and 20) of AlN_p and their metallurgical and mechanical properties have been analysed. Macrohardness, microhardness, Yield strength and ultimate tensile strength of the composite have improved with the addition of AlN_p in the Al matrix. Optical and SEM micrographs reveal the homogeneous distribution of reinforcement particles in the matrix and X-ray diffraction patterns ensure the dispersion of AlN_p reinforcement in AA6061 matrix.

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1. Introduction

In day to day life, usage of aluminium matrix composites (AMCs) is enormously increasing due to its high strength-to-weight ratio and enhanced mechanical and thermal properties. AMCs have become very attractive for automotive and aerospace applications [1–4]. Thus particulate form of reinforcement present in the composites exhibits an isotropic property when compared with other geometries of reinforcement such as fibre and flake. Fibre and flake composites are generally anisotropic. The advantages of particulate composites are improved strength, increased operating temperature, oxidation resistance, easy to produce, etc. [5].

Al–SiC and Al–AlN $_{\rm p}$ composites are widely used in microelectronic devices. Although the thermal conductivity of AlN (175 W/m K) is less than SiC (250 W/m K), AlN is chemically more stable than SiC. Aluminium does not react with AlN [6] whereas in Al–SiC composites Al $_{\rm 4}$ C $_{\rm 3}$ phase is formed by the reactions of Al with SiC and degrades the mechanical properties of Al–SiC composite [7,8]. AlN has good compatibility with Al alloy, excellent thermophysical properties, good interfacial adherence without interfacial reaction [6], high specific strength & stiffness, high thermal conductivity, high electrical resistivity, low dielectric constant

and tailorable coefficient of thermal expansion. Thus $Al-AlN_p$ composite is an ideal candidate material for electronic packaging materials [9,10]. AA6061 alloy is used as matrices, as they offer good thermal conductivity, low density, easy to process, good castability and weldability compared with other high thermal conductivity matrix metal like copper [11].

AlN_p reinforced aluminium matrix composite is fabricated through pressure infiltration of liquid aluminium [12,13], powder metallurgy [14], powder injection moulding process [15], squeeze casting technique [7,8,10], in situ reaction [16-18] and stir casting method [19,20]. Even though AMCs are fabricated by several methods as mentioned above, the stir casting method is currently employed commercially. Advantages of stir casting method include simple, flexible, economical, suitable for mass production and production of complex profiled composite components without damaging the reinforcement particles. Due to these salient features of stir casting method, recently many attempts have been made to produce different composites using this method [19-23]. Some of the difficulties encountered in the fabrication of AMCs in stir casting method are achieving a uniform distribution of the reinforcement particles in the matrix, wettability between the matrix alloy and reinforcement particles, porosity in the cast AMCs and chemical reactions between the reinforcement particles and matrix alloy.

Homogenous distribution, wetting of reinforcement particles with the matrix and minimized porosity in the cast can be achieved by choosing appropriate process parameters such as molten metal temperature, uniform feed rate of reinforcement

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Table 1 Chemical composition of AA6061-T6 alloy matrix.

| Element | Mg | Si | Fe | Mn | Cu | Cr | Zn | Ni | Ti | Al |
|---------|-----|------|------|-----|------|------|------|------|------|---------|
| wt% | 0.9 | 0.64 | 0.26 | 0.1 | 0.21 | 0.05 | 0.04 | 0.02 | 0.01 | Balance |



Fig. 1. The manufactured typical $AA6061-AlN_p$ composites.

particles, incorporation time and stirring speed [23,24]. Nassaj et al. [19] stated that by adding 2% of Mg, incorporation time was reduced and wettability of the AlN_p with Al melt was improved by which the mechanical properties of the $Al-AlN_p$ composite were increased.

Limited research work has been carried out on the mechanical and the metallurgical properties of stir cast Al–AlN $_{\rm p}$ composites containing wide range of reinforcement. In this paper, the metallurgical and the mechanical characterization of AA6061–AlN $_{\rm p}$ composite (by varying AlN $_{\rm p}$ range from 0% to 20% by weight) produced in an inert atmosphere using a modified stir casting technique with the bottom pouring arrangement are presented.

2. Experimental procedure

A cleaned extruded aluminium alloy (AA6061-T6) rods of 25 mm diameter were placed inside the stainless steel crucible of electric stir casting furnace with bottom pouring attachment. The chemical composition of AA6061 rods is presented in Table 1. In order to avoid any contaminations at high temperatures, inner side of the stainless steel crucible and stainless steel stirrer were coated. Temperature of the electric furnace was set to $1000\,^{\circ}\text{C}$. When the temperature of the furnace reached $650\,^{\circ}\text{C}$, argon gas was supplied into the crucible at a constant flow rate of 2 lpm to avoid the reaction of matrix with the atmospheric air. By the time the temperature reached $1000\,^{\circ}\text{C}$, 2% magnesium of total weight of

the composite to be produced was added to the melt to increase the wettability of AlN_p with Al alloy [19,20]. A stainless steel stirrer coupled with electric motor was immersed into the molten aluminium alloy and stirred at a constant speed of 450 rpm to form a vortex. Stirring was carried out to facilitate both incorporation and uniform distribution of the reinforcement (AlN_p) in the molten aluminium alloy [20]. Proper stirring speed is necessary to avoid excessive gas content which will form due to over agitation of melts and lead to unacceptable porosity in the cast [24].

A predetermined quantity of preheated AlN $_p$ at 750 °C (of size 3 to 4 μ and purity of more than 99.6%) was added into the molten matrix at the side of the vortex. AlN $_p$ particles were incorporated into the melt for 260 s. The mixture of molten aluminium and AlN $_p$ were further stirred for 1200 s before pouring into a preheated permanent mould at 300 °C through the bottom pouring arrangement. Argon was supplied until the entire melt was poured into the permanent mould. The composites were allowed to solidify in atmospheric air and were taken out from the mould after solidification. Similarly AA6061–AlN $_p$ composites containing different weight percentages of (ranging from 0 to 20) AlN $_p$ were produced. The manufactured AA6061–AlN $_p$ composites are presented in Fig. 1.

Specimens of required size were cut from the cast composite to carry out the metallurgical and the mechanical characterization. Standard metallographic procedure was adopted to prepare the specimens for optical, SEM and EDAX analysis. SEM and EDAX analysis were carried out by using a scanning electron microscope

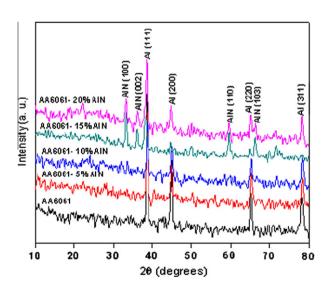


Fig. 3. The XRD patterns of fabricated AA6061–AlN_p composites.

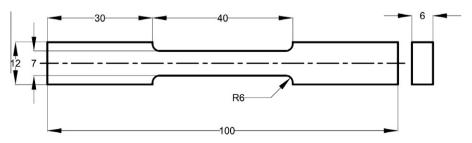


Fig. 2. The dimensions of the tensile specimen as per ASTM E8 M-04 standard.

(SEM) attached with energy dispersive spectroscope (JEOL-JSM-6390). X-ray diffraction patterns (XRD) were recorded using a Panalytical X-ray diffractometer. The polished specimens were etched with colour etchant containing 1 g of sodium hydroxide (NaOH) and 4 g of potassium permanganate (KMnO₄) in 100 ml distilled water for metallographic study. The microstructure was observed using an optical microscope (OLYMPUS-BX51 M) and photomicrographs of composites containing various weight percentages of reinforcement are presented in Fig. 4a–f.

The macrohardness was measured using a Brillnell hardness tester (model 7KB3000) at a load of 500 kg applied for 15 s at eight different locations on all samples. The microhardness of the etched AA6061–AlN_p composites was measured using a microhardness tester (MITUTOYO-MVK-H1) at a load of 500 g applied for 15 s at twenty different locations on all samples. The average values of macrohardness and microhardness were recorded. Three tensile specimens as per ASTM E8 M-04 standard as shown in Fig. 2 were prepared from each (% of reinforcement) cast composite [25]. The ultimate tensile strength (UTS) was estimated using a Computerized Universal Testing Machine (HITECH TUE-C-1000) and its average values were recorded.

3. Results and discussions

AA6061 alloy matrix reinforced with ${\rm AlN_p}$ composites were successfully fabricated by using the modified stir casting method with bottom pouring arrangement. The metallurgical and the mechanisms

cal characterization of fabricated AA6061–AlN $_{\rm p}$ composites are discussed below.

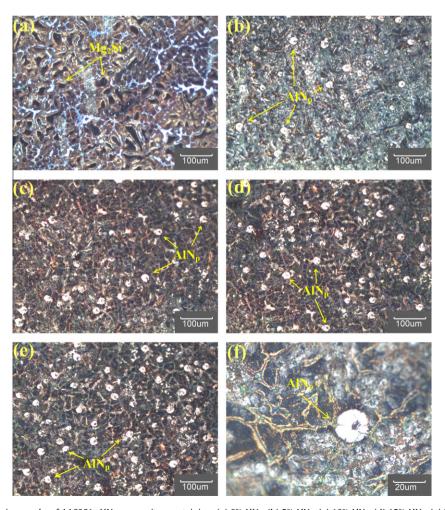
3.1. X-ray diffraction analysis of A6061–AlN_p composites

Fig. 3 shows the XRD patterns obtained from the fabricated composites which reveal the presence of AlN_p . The peaks of AlN_p are distinctly clear and they increase with the increase in AlN_p content while the peaks of AA6061 decrease. It is also interesting to note that the peaks of AA6061 in the composite are slightly shifted to lower 2θ when compared to that of AA6061 alloy. The XRD patterns also indicate that there is no formation of intermetallic compounds resulting from the reaction of AlN_p with AA6061 alloy matrix. From the XRD patterns, it is clear that AlN_p are thermodynamically stable at a fabrication temperature of 1000 °C.

3.2. Evaluation of microstructure of AA6061-AlN_p composites

Fig. 4 shows the microstructures of fabricated AA6061 alloy matrix as well as AA6061 alloy matrix reinforced with AlN_p composites. Microstructure of cast AA6061 alloy matrix is presented in Fig. 4a which reveals the formation of α -aluminium dendritic network structure which is formed due to the supercooling of composite during solidification. Precipitation of Mg_Si is also visible in the microstructure.

Fig. 4b–f shows the microstructure of AA6061–AlN $_{\rm p}$ composites containing different weight percentages of AlN $_{\rm p}$ reinforcement. Microstructures of the composites presented in Fig. 4b–e clearly



 $\textbf{Fig. 4.} \ \ \text{The optical photomicrographs of AA6061-AIN}_p \ composites \ containing: (a) \ 0\% \ AIN}_p, (b) \ 5\% \ AIN}_p, (c) \ 10\% \ AIN}_p, (d) \ 15\% \ AIN}_p, (e) \ 20\% \ AIN}_p, and (f) \ 5\% \ AIN}_p, (e) \ 20\% \ A$

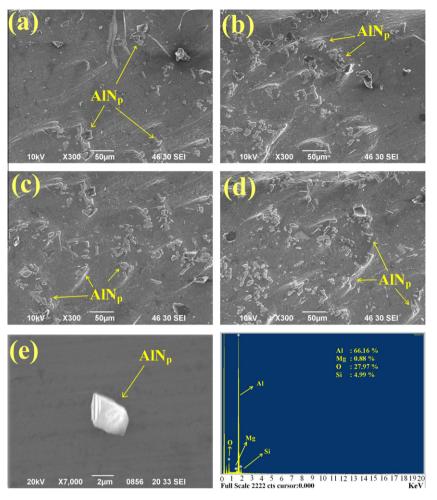


Fig. 5. The SEM micrographs of AA6061–AlN_p composites containing: (a) 5% AlN_p, (b) 10% AlN_p, (c) 15% AlN_p, (d) 20% AlN_p, (e) 5% AlN_p, and (f) EDAX analysis of AA6061–AlN_p composites containing 20% AlN_p.

reveals the homogeneous distribution of the AlN_p in the Al alloy matrix and there is no evidence of porosity and cracks in the castings. This might be related to proper process parameters employed for the production of castings. During solidification of AA6061– AlN_p composite, AlN_p are rejected in the direction of refined α -Al grains. Refinement of α -Al grains may be due to AlN_p themselves, which act as nucleus on which the α -aluminium grains solidify and AlN_p offer resistance to the growing α -Al phase during the solidification process. Fig. 4c shows the precipitation of Mg_2Si . The sources for the formation of Mg_2Si are addition of Mg in molten Al alloy matrix and Mg and Si are already present in the AA6061 alloy as major constituent.

From the SEM micrographs, it is clear that ${\rm AlN_p}$ are clustered in a uniform manner and distributed homogeneously in the matrix. Hence, appearance of single particles in the optical photomicrographs contain cluster of ${\rm AlN_p}$. It is also evident from the photomicrographs that cluster of ${\rm AlN_p}$ in the matrix are spherical shape. Fig. 4f shows the clear interface between the Al alloy matrix and ${\rm AlN_p}$ reinforcement.

A small amount of magnesium aluminate (spinel) $MgAl_2O_4$ is formed in the matrix during the process according to the following reaction [19].

$$3Mg + 4Al_2O_3 \rightarrow 3MgAl_2O_4 + 2Al \tag{1}$$

Formation of magnesium aluminate is confirmed in the EDAX analysis which is presented in Fig. 5f. Source of oxygen for the above reaction is the oxide layer formed over the molten Al alloy

matrix. Since an inert atmosphere is maintained at the top of the molten matrix, constant motorized stirring action is carried out during the incorporation of AlN_p in the matrix and molten metal is poured through bottom of the crucible, there is no formation of magnesium aluminate at the interface of matrix and AlN_p reinforcement.

Fig. 5a–d shows the SEM micrographs of fabricated AA6061–AlN $_{\rm p}$ composites containing 5% AlN $_{\rm p}$, 10% AlN $_{\rm p}$, 15% AlN $_{\rm p}$ and 20% AlN $_{\rm p}$ respectively. SEM micrographs show the uniform distribution of AlN $_{\rm p}$ reinforcement in the AA6061 alloy matrix. The SEM micrograph presented in Fig. 5e reveals a clear interface between AA6061 alloy matrix and AlN $_{\rm p}$, which is due to the absence of interfacial reaction between matrix and the reinforcement. The addition of magnesium enhances the wettability between AlN $_{\rm p}$ and AA6061 alloy matrix which results in good bonding between AlN $_{\rm p}$ and AA6061 alloy matrix.

3.3. Evaluation of mechanical properties

Fig. 6 shows the average macrohardness and microhardness of AA6061–AlN $_{\rm p}$ composites. The macrohardness of AA6061–20% AlN $_{\rm p}$ composite is 79 BHN which is 107.89% greater than AA6061 matrix. Similarly microhardness of AA6061–20% AlN $_{\rm p}$ composite is 91 VHN which is 106.81% greater than AA6061 matrix. Both macro and microhardnesses of AA6061–AlN $_{\rm p}$ composites linearly increase with the addition of AlN $_{\rm p}$ due to the presence of hard AlN $_{\rm p}$ in AMCs.

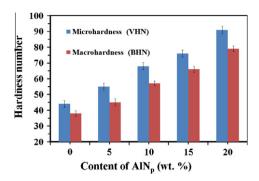


Fig. 6. Effect of weight percentage of AlN_p on microhardness and macrohardness of $AA6061-AlN_p$ composites.

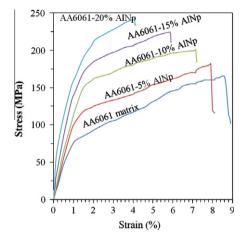


Fig. 7. Effect of weight percentage of AlN_{p} on stress strain curve of $\text{AA6061-AlN}_{\text{p}}$ composites.

Fig. 7 reveals the typical engineering stress strain diagrams of AA6061 matrix and AA6061–AlN $_{\rm p}$ composites obtained by tensile test conducted at room temperature. The average Yield strength

measured at 0.2% offset, UTS and % elongation of AA6061-AlN_p composites with various wt% of AlN_p are presented in Fig. 8. Both Yield strength and UTS are increased by the addition of AlN_p. But % elongation of the AMC decreases when the amount of AlN_p increases. Yield strength of AA6061-20% AlN_p composite is 158 MPa which is 95.12% greater than AA6061 matrix. UTS of the AA6061-20% AlN_p composite is 241 MPa which is 46.95% greater than AA6061 matrix alloy. An increase in volume fraction of low coefficient of thermal expansion reinforcement material in the higher coefficient of thermal expansion matrix changes the microstructural characteristics of the metal matrix with a concomitant contribution to increasing strength. As such, an increase in the volume fraction of the reinforcing ceramic particulate results in an increase in dislocation density around the reinforcement particles during solidification [26], reduction in grain size, and an overall reduction in the substructure. The above microstructural changes tend to increase the resistance offered to the motion of both microscopic and macroscopic level dislocations under the influence of a far-field stress [27]. Accordingly, an increase of wt% of AlN_p reinforcement in the AMCs increases the magnitude of resistance offered to the motion of both microscopic and macroscopic level dislocations. As a result macrohardness, microhardness and UTS increase with the increase in wt% of AlN_p reinforcement. Clear interface noticed between the matrix and AlN_p reinforcement and uniform distribution of AlN_p reinforcement increase the load bearing capacity of the composite [28].

The enhancement in UTS is, however, correlated with a reduction in ductility, which reduces to about 4.1% for the AA6061–20% ${\rm AlN_p}$ composite, as apparently shown in Fig. 8c. Increasing the wt% of ${\rm AlN_p}$ in the composite resists the flowability of aluminium matrix and reduces the ductile aluminium alloy matrix content [26] which results in the decrease of % elongation of the composite. The mechanical properties of AA6061 matrix and AA6061 matrix reinforced with different weight percentage of ${\rm AlN_p}$ are summarized in Table 2. It is evident that the incorporation of ${\rm AlN_p}$ into AA6061 matrix significantly improves the strength of the composites.

Fig. 9a reveals the fracture surface of AA6061 matrix alloy. It shows a net work of large size dimples which indicate large

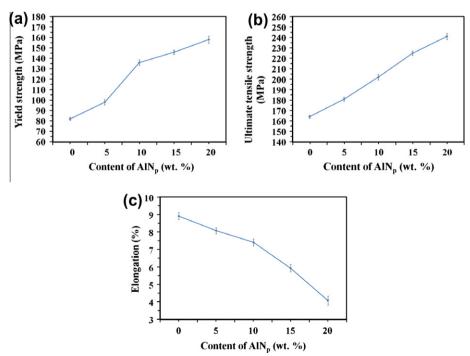


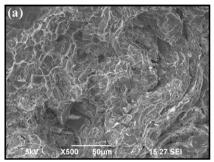
Fig. 8. Effect of weight percentage of AlN_p on (a) Yield strength, (b) ultimate tensile strength, and (c) % elongation of AA6061-AlN_p composites.

Table 2The mechanical properties of AA6061–AlN_p composites.

| S. No. | Material | 0.2% Yield strength (MPa) | | UTS (MPa) | | Microhardness (VHN) | | Macrohardness (BHN) | | % Elongation | |
|--------|-----------------|---------------------------|------------|-----------|-----------|---------------------|-----------|---------------------|-----------|--------------|-----------|
| | | Ave.a | Std. dev.b | Ave. | Std. dev. | Ave. | Std. dev. | Ave. | Std. dev. | Ave. | Std. dev. |
| 1 | AA6061 matrix | 82 | 1.53 | 164 | 1.53 | 44 | 2.15 | 38 | 1.70 | 8.91 | 0.20 |
| 2 | AA6061-5% AlNp | 98 | 2.52 | 181 | 2.08 | 55 | 2.16 | 45 | 2.22 | 8.07 | 0.17 |
| 3 | AA6061-10% AlNp | 136 | 1.53 | 202 | 2.65 | 68 | 2.27 | 57 | 1.43 | 7.40 | 0.21 |
| 4 | AA6061-15% AlNp | 146 | 2.08 | 225 | 2.08 | 76 | 2.22 | 66 | 1.99 | 5.93 | 0.21 |
| 5 | AA6061-20% AlNp | 158 | 3.46 | 241 | 3.06 | 91 | 2.20 | 79 | 1.88 | 4.07 | 0.26 |

^a Average value.

b Standard deviation.



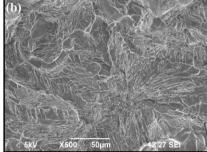


Fig. 9. Fracture surface of (a) AA6061 and (b) AA6061-20wt% AlN_p.

amount of plastic flow prior to failure. Fig. 9b reveals the fracture surface of AA6061–20% $\rm AlN_p$ composite. It shows a net work of dimples whose size is smaller compared to matrix alloy. The $\rm AlN_p$ refined the grain size of matrix alloy and reduced the ductility which resulted in smaller size dimples.

4. Conclusion

The $AA6061-AlN_p$ composites containing different weight percentage of reinforcement viz 0%, 5%, 10%, 15% and 20% were successfully produced by stir casting method. The conclusions derived from the research work carried out are given below.

- The homogeneous dispersion of AIN_p in AA6061 matrix alloy is present.
- The good bonding and clear interface between the AlN_p reinforcement and the aluminium alloy matrix are existed.
- XRD patterns ensure the presence of $\mbox{AlN}_{\mbox{\scriptsize p}}$ in the composites.
- $-\,$ UTS of the AA6061–20% AlN_p composite is 46.95% greater than the corresponding monolithic alloy.
- Higher the percentage of AlN_p reinforcement in the matrix, higher is the macrohardness and microhardness of the composite but lower is the % of elongation of the composite.

Acknowledgements

The authors wish to place their sincere thanks to the Management and Department of Mechanical Engineering, Coimbatore Institute of Technology, Coimbatore, India for extending the facilities of Welding Research Laboratory to carry out this investigation. The authors wish to register their sincere thanks for financial support rendered by Naval Research Board, DRDO, Govt. of India, vide funded project; Ref. No. DNRD/05/4003/NRB/85 dt. 30.10.2006. Authors also wish to thank Karunya University, Coimbatore, India for provid-

ing SEM, UTM testing facilities. Authors are also thankful to Mr. S.J. Vijay, Mr. I. Dinaharan, Mr. K. Kalaiselvan, Mr. M. Thambidurai, Mr. V. Rajasekaran, Mrs. V. Sarala Devi and Mr. A. Raja for their assistance offered to execute the above work.

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